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PROCESS IDENTIFICATION STUDY FOR SPACE CURED COMPOSITE STRUCTURES

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1.0 INTRODUCTION

This report summarizes the findings of a literature study on space cured composite structures and recommends a course of future action. The information contained herein was collected from the following sources:

Chem Abstracts	1956-present
Engineering Index File	1970-present
NTIS	1964-present
COMPENDEX	1964-present
LMSC Inhouse File (over 178,000 catalogued books and reports)	1955-present
NASA Search	1968-present
DDC Search	1967-present

In addition a number of interviews were conducted to add further substance to the literature search. NASA/JSC, NASA/MSFC, and AFML were consulted because of past and present interests in this approach. Shell Chemical and Dow Chemical were quizzed on new matrix approaches, particularly the epoxy acrylates which combine epoxy properties with polyester type curing.

All of the literature references have been reviewed and a complete bibliography appears at the end of this discussion. The key to the report is a comprehensive table on materials and approaches for space cured composite structures. Following the table is a discussion of the most widely used approaches and materials along with comments on their viability.

Most of the applications used for demonstration of the various candidate materials and techniques involved the fabrication of solar collectors, space shelters and docking stations. In one or two cases, however, techniques were

used which could be applied to the fabrication of inflatable, space cured tubular structures. The primary matrix materials involved were gelatin, polyurethane, epoxy and unsaturated polyester. Other systems were given minimum consideration and with one exception probably will not be involved in any future work. Each of these topics is a subsection of the discussion. By far the most glaring omission is lack of data on the use of the various matrices with graphite fiber.

The approaches used to cure the various resins cited in the literature are discussed with each matrix material. The literature revealed the following curing techniques were used to rigidize the structures:

- o Plasticizer or solvent boil-off
- o Vapor reactions and/or catalysis
- o Ultraviolet radiation
- o Thermal cure
- o Elastic memory
- o Anaerobic cure

At the end of the discussion section a number of conclusions are made as to the viability of various materials and approaches. Included are suggested areas for future work. Following this are recommendations for further study prioritized in accord with the authors' feelings as to which procedures and materials have the best chance of success.

Early in the program a set of selection criteria were developed as an aid in the evaluation of the various approaches to space cured composite structures. Factors pertaining to both the fabrication and the mechanical properties of the resultant structures were considered. The criteria that were used follow:

Factors Pertaining to Cure and Fabrication

1. Tube must remain flexible during expansion
2. Cure should proceed at reasonable rate under ambient space conditions
3. Approach must provide for positive fiber alignment

Factors Pertaining to Cure and Fabrication (cont'd)

4 System must be packageable with regard to weight, volume, tack, etc.

5 Material must possess adequate shelf life

6 Process must be safe with regard to toxicity, explosion hazard, etc.

7 Chemistry must provide for low void content

8 Internal stresses resulting from shrinkage during cure should not compromise tube performance

Factors Pertaining to Physical/Mechanical Properties

9 Material must be stable in space environment (vacuum, UV, radiation) for intended lifetime of the system

10 Matrix modulus must be in the glassy region ($>10^9$ pa) over the temperature range of interest

11 Adequate strength must be maintained over the temperature range of interest

12 Composite should possess zero or low coefficient of thermal expansion

2.0 DISCUSSION

Shown on the following page is a table which summarizes the results of this investigation. Specific matrix systems and the ways in which they rigidize and cure are listed along with the above stated selection criteria which investigators have addressed and found adequate. Possible problems associated with each matrix system are outlined and references referring to one or more of the chart headings are delineated.

2.1 Gelatin Type Technology

The first category of matrix resins discussed includes those that ridigize by plasticizer or solvent boil-off. These resins do not chemically react but simply harden as they evaporate to dryness in a space environment. Gelatin has received the most attention because of several very attractive features. It is derived from abundant natural sources and should be quite economical. Flexural properties of glass laminates are good up to 250^{o}F (121 K) and the environmental resistance to UV, IR and gamma radiation appears adequate. The main advantage to this approach is the reversible nature of the plasticizer which permits structural elements to be deployed and tested on the ground prior to packaging and flight. The primary disadvantage is the weight penalty of about 10-15% (6,43) incurred by the weight of plasticizing liquid (water) eventually lost in space. Since most of the work was done with glass fabric, compatibility with graphite is unknown. It is not known how well unidirectional fiber alignment can be maintained during rigidization or how internal stresses arising from shrinkage affect long term properties.

Much less work has been done with the other resins in this category which include polyvinyl alcohol (PVA), poly (hydroxyethyl methacrylate) [Hydron] and phenoxy. PVA requires about 23% water for plasticization and Hydron 13-14%

SUMMARY OF MATERIALS AND APPROACHES FOR SPACE CURED COMPOSITES

MATRIX SYSTEMS	RIGIDIZING PROCESS	CURE MECHANISM	SELECTION CRITERIA MET		POSSIBLE PROBLEMS	REFERENCES
			FABRICATION	PERFORMANCE		
Gelatin	Plasticizer boil-off	Physical	1,2,4,5,6,7	9,10,11	Weight penalty, high shrinkage graphite fiber compatibility	7,9,10,14,15,16, 27,28,33,35,43
PVA	Solvent boil-off	"	1,2,5,6,7	10,11	" " "	6
Hydron	Plasticizer boil-off	"	1,2,5,6,7	10,11	" " "	6
Phenoxy	Solvent boil-off	"	1,2,5	10,11	" " "	6
Urethane	Vapor cure	H_2O+NCO	1,2,4,6	9,10,11	Packaging, forming, blocking, fiber orientation	2,5,7,9,12,14, 15,17,18,19,23, 24,25,26,28,30, 32,34,35,39,43
Polyester	Vapor cure by tertiary amine	Accelerated peroxide,peroxide accelerated by t-amine	1,2,4,5,7	9,10,11	Blocking, cure rate, fiber orientation	2,5,9,22,23, 31,37,41,42
	UV cure	Light+accel.-peroxide	1,2,4,5,7	9,10,11	Cure with graphite, blocking, fiber orientation, uniform cure	4,9,13,15,22, 28,38,40,41,42, 43,44
	IR (thermal)	Peroxide, free radicals	1,2,4,5,7	9,10,11	Blocking, fiber orientation, uniform cure	45
	Anaerobic	Loss of oxygen inhibitor	1,2,4	10	Blocking, fiber orientation	3
Epoxy	Vapor cure	Amine + resin	1,2,4,5,7	9,10,11	Blocking, fiber orientation, cure rate	11,24,31,34, 37,43,44
	Thermal cure	Amine + resin	1,2,4,5,6,7	9,10,11	Blocking, fiber orientation, heat source	18,22,38,40, 41
	Elastic Memory	Amine + resin	1,2,3,4,5,6 7	9,10,11	Heat source, resin elongation, blocking	29,36
n-Vinyl Carbazole (NVC)	Vapor cure	Volatile acid	1,2,4,5,7	10,11	Physical form of matrix, blocking, toxic, corrosive vapors	44,46
Cyclized Polybutadiene	Thermal cure	Peroxide, free radicals	2,3,4,5,6 7	10,11	High temperature cure	46
NVC+CPBU	Vapor cure	Heat liberated from NVC cures CPBU			Inadequate cure	46
Furfuryl Alcohol	Vapor cure	Volatile acid			Blocking,adequate cure, physical properties, corrosion, toxicity	6,37
Phenyl Silane	Vapor cure	Unknown			High pressure, high temperature cure	37
Acetoxy Silane	Vapor cure	Unknown			Largely unexplored	1

2.1 Gelatin Type Technology (cont'd)

and they soften at lower temperatures than gelatin., i.e., 185°F (85 K) and 170-200°F (77-93 K), respectively. Phenoxy was dissolved in methylethylketone (MEK) which may be hazardous due to its low flash point. Phenoxy's heat distortion temperature is given as 212°F (100°K). A polyacrylic modified with a styrene malaic copolymer and melamine was investigated (34) but not developed to fruition.

2.2 Urethanes

A great deal of work has been done with water vapor cured polyurethanes. Fiberglass laminates cured in 15-30 minutes exhibit flexural strengths comparable to those of epoxies. Large structures including cylinders 1.07m diameter and 1.2m long, a space shelter 2.1m in diameter and 2.4m high and a semi-cylindrical 4 by 4.6m quonset hut were fabricated from water cured polyurethanes. Smaller structures can be inflated from residual vapor pressure alone while larger structures require auxiliary inflation equipment. Fluted fiberglass reinforcement was used for these demonstrations. The main drawback of polyurethanes concerns shelf life. Stringent precautions must be taken to isolate the prepreg from moisture to prevent premature hardening. This necessitates impermeable packaging which introduces a weight penalty and a disposal problem. Available literature indicates only slight degradation of flexural modulus for two urethane resins when exposed to IR and UV radiation over the course of 4 weeks. The effects of longer exposure are unknown. The chemistry of polyurethanes cured in this manner evolve carbon dioxide which can cause foaming, depending on the rate of reaction. It appears, however, that this concern is not as serious as anticipated.

2.3 Polyesters

Polyesters cured by vapor catalysis, UV radiation or IR (heat) have been investigated for rigidizable space structures. Mechanical properties are adequate but degradation due to continuous exposure to UV and gamma radiation is suggested by the limited data on hand (2). UV cures are very fast and deployment times may not be sufficient enough to assure dimensional stability

2.3 Polyesters (cont'd)

prior to rigidization. It should be noted that in a discussion with Hexcel they stated they were unable to obtain UV cures in graphite composites.

Shrinkage is rather high with polyesters in general and high internal stresses could develop if adequate measures are not taken to insure uniform exposure during cure. High shrinkage combined with poor adhesion in general point to loss of strength with aging. IR cure also requires uniform exposure to the sun and resins conducive to this type of cure remain warm during service, thereby functioning with reduced strength (43). Gas catalysis appears to be the most viable approach for polyesters. However, this approach requires special packaging containers and the associated weight penalty.

2.4 Epoxy Resin Technology

Three rigidizing methods for cured in space epoxy matrix composite structures have been studied; vapor cure, thermal cure and elastic memory. Of the three methods, vapor curing has apparently received the most attention. Numerous catalysts and curing agents have been tried including Lewis bases such as tertiary amines and triphenylphosphine and Lewis acids such as stannic chloride and diamines such as 1,3-propylene diamine and 1,4-cyclohexane bis (methyl amine). Surprisingly, the use of ammonia as a curing agent could not be found in the literature. In our interviews, Dr. R. W. Tess of Shell Chemical stated that he was able to cure surface coatings up to 10 mils in thickness using this technique. It was claimed that poly (allyl glycidyl ether) gave excellent coatings.

Another technique that received considerable attention was that of heat curing systems. This approach has the advantage of being able to choose from a wide variety of curing agents but this can cause shelf life problems. Probably the best system for this purpose would be a "kicked dicy" formulation. These systems have almost infinite pot life and cure around 246°F (119 K). We have calculated that using resistance heating (wires in the composite) the power required to cure a 15.2cm diameter tube 0.76mm thick and 9.1m long at 250°F (121 K) in space, in the absence of sunlight, would require a solar cell power

panel area of 46 square meters. Perhaps a weight penalty could be avoided by using the graphite fibers as the resistance element.

Another technique which was mentioned briefly in the literature is that of elastic memory. One of the authors holds patent rights on this concept involving epoxy resins. Elastic memory is the ability of a deformed thermoset structure to regain its original shape.

At its glass transition temperature (Tg) a thermoset matrix has a low modulus and displays high elongation to break. Under these conditions it can be pressed into another shape and if held in this condition during cooling it retains the strained, collapsed shape indefinitely. However, when reheated above its Tg the structure regains its original shape without external force. Thus a tubular structure could be collapsed and reformed in space simply by heating. One article was found in the literature indicating the feasibility of the approach. The concept was demonstrated using glass fibers embedded in an epoxy matrix to form a collapsible aircraft wing tank for packaging during transport. The idea has not been considered for space applications. This could be a high payoff area.

Our visits to Shell and Dow revealed an interesting new raw material in the epoxy acrylates (trade names EPOCRYL and DERAKANE). These materials are available in a variety of forms and cure like a polyester but have epoxy type properties. Accordingly, the rigidizing technology used would be identical with that discussed in the previous section under Polyesters.

From the standpoint of structural integrity and proven characteristics with graphite fibers the epoxies as a class are ideal candidates. In addition to that of selecting the curing agents the major problem appears to be one of blocking (the tendency of adjacent layers of prepreg to stick to each other) which complicates high density packaging concepts. It is, of course, possible to use barrier films to prevent blocking but this complicates the manufacturing process. There are other possible solutions to this problem. One is to

partially react the product to a non-tacky, rubbery gelled state and use a second curing agent to complete the process. Another is to incorporate an elastomer in the formulation in sufficient quantity to eliminate the tack and harden the tube with a second curative. A third approach would be to chemically react the outer surface of the composite layup. For example, the epoxy acrylates contain a plurality of hydroxyl groups on the backbone structure which might be used in conjunction with a diisocyanate to form the surface skin.

2.5 Miscellaneous Resin Systems

The most promising matrix system which falls under none of the above categories is a cyclized polybutadiene urethane (CPBU) developed by TRW under an LMSC subcontract(46). The resin displays good compressive and flexure strengths up to 500°F (260 K) after thermal cure at 350°F (177 K). While this particular resin requires extensive thermal energy (4 kw-min/sq ft for prepreg on fluted glass fabric) in order to reach 350°F (177 K),the method offers the following distinct advantage. The matrix resin is prepregged and then B-staged (partially cured) to a flexible non-tacky, rubbery state which provides excellent packaging characteristics - no shelf life problem and no weighty impermeable containers. In addition, the fiber alignment is maintained by the rubbery state. By following a similar B-staging approach with a 250°F (121 K) or lower curing resin on graphite one might circumvent the current deficiency, especially if the graphite fibers could be used for resistance heating.

N-vinyl carbazole showed some promise as a 93-121 K matrix system although the gas required for catalysis, viz. dinitrogen tetroxide,is highly corrosive and toxic. There is an added complication in that N-vinyl carbazole is a crystalline solid. A combination of N-vinyl carbozole and CPBU was found to be unworkable.

Furfural alcohol cured with HCl gas shows no promise due to slow cures and poor strengths coupled with the highly toxic and corrosive nature of the

2.5 Miscellaneous Resin Systems (cont'd)

catalyst. A vapor cure for a modified phenyl silane was briefly investigated until it was realized that high temperatures and pressures were required for suitable fabrication. An acetoxy silane was under development when Air Force funding limitations apparently cut this effort short.

3.0 CONCLUSIONS

The most widely studied matrix resin appears to be gelatin. It exhibits good mechanical properties to 121K and space environmental exposure data (UV,IR,gamma radiation) looks good. A strong selling feature is the reversibility which permits terestial quality control. The main deficiencies are the weight penalty of plasticizer (water) and substantial shrinkage stresses which may prove troublesome upon aging. No data are available with graphite fibers. Any future work should address the potential shrinkage problem and generate data on graphite fibers.

Water cured polyurethanes have received considerable attention and large scale demonstrations have been successful. Properties are quite satisfactory. Perhaps the biggest drawback is the stringent requirement that water vapor be excluded during storage. Therefore a barrier weight penalty is exacted in addition to the weight of water required for cure.

Polyesters have received less attention than either of the above systems. UV cured polyesters require a uniform exposure during cure and will not necessarily work with graphite fibers.. However, amine vapors can be used to initiate cure of peroxide catalyzed systems. With the advent of newer systems such as the epoxy acrylates, superior properties could be realized. This area warrants further investigation.

Epoxy resins have not really received adequate evaluation in light of their proven properties as matrix resins for graphite reinforced composites. A recent visit to Shell Development Company in Houston, Texas, has directed our attention to considerable success which they have had with ammonia vapor cures of epoxy systems. Utilization of this approach was not found in our cured-in-space literature search.

A cyclized polybutadiene urethane, while not desirable as a material per se, was interesting from a processing standpoint. B-staging a resin to a non-tacky, rubbery state offers a weight savings in terms of eliminating separator films and provides for positive fiber alignment during deployment and cure.

The "elastic memory" approach demonstrated for an epoxy formulation but applicable to other materials as well holds considerable potential. The technique can be reversible, provide for positive fiber alignment and provide desirable packaging characteristics while offering satisfactory physical/mechanical properties. Although this approach may be considered high-risk/high payoff, these features provide high reliability and ease of handleability unattainable by other approaches. Since a well controlled heating rate is not as critical for the elastic memory approach as for one involving chemical cure a solar concentrator may offer adequate energy for deployment.

4.0 RECOMMENDATIONS FOR FUTURE EFFORT

Listed below are a series of general concepts which should be considered for future work. They have been prioritized based on the author's analysis of the data available, the topmost idea being considered as the best.

- The elastic memory concept should be studied further. Although this is a high risk approach the rewards appear great. An epoxy/graphite system is the recommended starting point.

- Develop the concept of non-tacky pliable composite layup in order to minimize packaging. Using epoxies and/or epoxy acrylates through chemical reaction, partial gelatin or elastomer inclusion make a non-blocking packageable system which can be subsequently cured to a rigid structure.

- Generate further data on the evaporative rigidization concept answering such questions as the effect of graphite fibers and the plausibility of other matrices of this type.

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